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# Physical and Chemical Properties of Respirable Coal Dust from Two United States Mines

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**This investigation was aimed at establishing procedures for obtaining data relative to the physical and chemical characterization of respirable coal mine dust for utilization in epidemiological studies of incidence of coal workers' pneumoconiosis in different United States mines. In this paper the dust sampling and analytical procedures are briefly described and results are presented for three samples of approximately 4 grams each collected in each of two United States coal mines representing two different coal seams. Analyses were performed to determine the extent of differences in dust properties between (1) samples collected in the same mine and (2) samples collected in each of the two mines.**

## Introduction

THE PURPOSE OF THIS PAPER is to illustrate an approach to coal mine dust characterization for epidemiological investigations. Data are presented relative to the physical and chemical properties of respirable coal mine dust collected in two mines representing two different seams in Western Pennsylvania. These data are part of a study concerned with sampling respirable coal mine dust in United States mines representing different seams associated with varying incidences of coal miners' pneumoconiosis. It is hoped that this study of the physical and chemical properties of the dust as well as the concentrations to which miners are exposed on a day-to-day basis will promote understanding of the degree and intensity of miner lung response following inhalation of the different coal dust clouds.

The background of the study and the details of methodology have been previously reported.[1] The details of sampling and analytical methodology will not be repeated here but a brief review of these methods is in order.

## Methodology

Samples are collected underground approximately 200 feet from the active face in the return airway. A horizontal elutriator was desinged fabricated and calibrated to operate at approximately 36 cfm for a full shift. Samples are collected on Millipore membrane filters Type SCW P00010 measuring 8 inches by 10 inches. The filter with sample is returned to the laboratory where the dust is subsequently scraped from the filter face by means of a razor blade. Two such samplers operating side by side are used to collect three samples of approximately 4 grams each in each mine tested. The 4-gram sample is a composite of material collected on several days of full shift sampling. The concentrations of respirable coal

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mine dust in the mine being sampled will determine the time period required to obtain the samples weighing 4 grams a quantity fixed by requirements for physical and chemical analyses of the samples.

A BAHCO centrifugal classifier is then used to separate the respirable dust sample into four particle size fractions. The particle sizes associated with each of the four graded cuts, designated as throttles 14, 16, 17, and 18, are approximately 5.5 to 11.0 μm, 2.8 to 5.5 μm, 1.6 to 2.8 μm, and less than 1.6 μm, respectively. All particle sizes are expressed as aerodynamic equivalent diameter, the diameter of a unit density sphere which settles in air at the same velocity as the particle.

The following physical tests are performed on each of the four particle size fractions.

*Density*

Density is determined by a liquid pycnometric method and by an air pyconometry method developed in our laboratory.[2]

*Particle Size Distribution by Optical Microscopy*

Phase-contrast microscopy is used in conjunction with a calibrated eyepiece graticule to determine the microscopic particle size distribution.

*Total Random Projected Area*

A light extinction apparatus is utilized to determine the projected area of the coal dust particles while they are in random orientation during suspension in water.

*Particle Size Distribution by Sedimentation in Liquid*

Sedimentation analysis is performed by utilizing a Cahn microbalance sedimentation column with approximately 30-mg samples. Inherent limitations of the sedimentation methodology have prevented our obtaining valid sedimentation analysis of the smallest two graded cuts from the BAHCO classifier. The comparison of particle projected area diameters by microscopy and hydrodynamic equivalent diameters by sedimentation yields and effective shape factor for the particles.

*Determination of Particle Total Surface Area*

The total particle surface area of samples

TABLE I
Results of Physical Analysis of Four Fractions of Respirable Coal Mine Dust Collected by Horizontal Elutriator in Mine M and Separated by BAHCO (Type M-2 Coal)

| BAHCO Throttle No. | 18 | 17 | 16 | 14 | Total Dust |
|---|---|---|---|---|---|
| Median size $d50_e$ by BAHCO (μm) | | | | | 3.66 |
| Lower quartile $d25_e$ by BAHCO (μm) | | | | | 2.26 |
| Upper quartile $d75_e$ by BAHCO (μm) | | | | | 5.99 |
| Median size $d50_p$ by microscopy (μm) | 2.14 | 3.80 | 6.40 | 8.70 | 4.10 |
| Lower quartile $d25_p$ by microscopy (μm) | 1.49 | 2.55 | 4.22 | 6.10 | 2.72 |
| Upper quartile $d75_p$ by microscopy (μm) | 2.90 | 5.20 | 7.50 | 12.00 | 5.55 |
| Median size $d50_s$ by sedimentation balance (μm) | | 4.00 | 5.28 | 6.80 | 6.82 |
| Lower quartile $d25_s$ by sedimentation balance (μm) | | 2.80 | 4.50 | 4.90 | 4.80 |
| Upper quartile $d75_s$ by sedimentation balance (μm) | | 6.30 | 6.50 | 8.20 | 8.30 |
| Random projected area by light extinction ($cm^2/gm$) | $6.14 \times 10^3$ | $6.13 \times 10^3$ | $4.67 \times 10^3$ | $2.98 \times 10^3$ | |
| Total surface area by BET ($cm^2/gm$) | $2.41 \times 10^5$ | $1.92 \times 10^5$ | $1.11 \times 10^5$ | $6.73 \times 10^4$ | |
| Density by air pycnometry ($gm/cm^3$) | 2.22 | 2.36 | 2.37 | 2.16 | |

is determined by adsorption analysis with krypton or nitrogen gas and application of the Brunauer, Emmett, and Teller (BET) method for calculating total specific surface area.

Particle size fractions from the BAHCO classifier are also subjected to chemical analysis. The following analyses are performed:

Ultimate analyses of samples by use of the Perkin-Elmer Model 240 carbon-nitrogen-hydrogen analyzer.

Free silica in ash analyses by infrared analysis.[3]

Trace metal analyses by means of a Perkin-Elmer Model 403 atomic absorption spectrophotometer with teletype hookup. The samples are prepared by a new technique which utilizes treatment by fuming nitric acid and dissolution of silaceous material by hydrofluoric acid digestion bomb.[4]

TABLE II

Results of Chemical Analysis of Four Fractions of Respirable Coal Mine Dust Collected by Horizontal Elutriator In Mine M and Separated by BAHCO (Type M-2 Coal)

| BAHCO Throttle No. | Substance | 18 | 17 | 16 | 14 | Total Dust |
|---|---|---|---|---|---|---|
| Free silica analysis (weight %) | $SiO_2$ | 7.3 | 7.8 | 7.2 | 5.5 | |
| | Ash | 63.5 | 56.6 | 45.4 | 39.1 | |
| Proximate analysis (weight %) | Moisture | | | | | |
| | Volatile matter | | | | | |
| | Fixed carbon | | | | | |
| | Ash | | | | | |
| Ultimate analysis (weight %) | Carbon | 22.6 | 29.4 | 39.0 | 42.6 | |
| | Hydrogen | 2.2 | 2.5 | 2.9 | 3.1 | |
| | Sulfur | | | | | |
| | Nitrogen | | | | | |
| | Oxygen | | | | | |
| | Ash | 65.0 | 57.9 | 47.3 | 42.3 | |
| Trace element analysis (weight %) | Aluminum | 5.75 | 5.20 | 3.10 | 2.08 | |
| | Barium | <0.05 | <0.05 | <0.05 | <0.05 | |
| | Beryllium | <0.05 | <0.05 | <0.05 | <0.05 | |
| | Bismuth | 0.13 | 0.13 | 0.07 | <0.05 | |
| | Cadmium | 0.003 | 0.003 | 0.003 | 0.002 | |
| | Calcium | 1.75 | 2.10 | 3.90 | 3.33 | |
| | Chromium | <0.01 | <0.01 | <0.01 | <0.01 | |
| | Cobalt | 0.03 | 0.03 | 0.04 | 0.03 | |
| | Copper | 0.40 | 0.20 | 0.15 | 0.09 | |
| | Iron | 1.43 | 1.28 | 0.90 | 0.75 | |
| | Lead | <0.04 | <0.04 | <0.04 | <0.04 | |
| | Lithium | 0.013 | 0.011 | 0.010 | 0.010 | |
| | Magnesium | 0.408 | 0.353 | 0.276 | 0.245 | |
| | Manganese | 0.03 | 0.02 | 0.02 | <0.02 | |
| | Nickel | 0.03 | 0.03 | 0.02 | <0.02 | |
| | Potassium | 0.86 | 0.73 | 0.45 | 0.41 | |
| | Silver | 0.03 | 0.03 | 0.04 | 0.03 | |
| | Sodium | 0.061 | 0.052 | 0.050 | 0.420 | |
| | Strontium | 0.09 | 0.08 | 0.07 | 0.06 | |
| | Vanadium | <0.05 | <0.05 | <0.05 | <0.05 | |
| | Zinc | 0.25 | 0.22 | 0.25 | 0.09 | |

## Results and Discussion

### *Variations in Physical and Chemical Properties of Respirable Dust Samples Collected in the Same Mine*

Before comparing properties of dust samples collected in different mines, it is necessary to know the extent of variation in properties among samples colletced in the same mine. For this purpose, three integrated respirable coal mine dust samples of approximately 4 grams each are collected in each mine. Each sample represents up to five shifts of sampling time in each of the mines M and R to be discussed in this paper. Mine M and mine R are in the Pittsburgh and Lower Freeport seams, respectively.

Tables I and II summarize results of physical and chemical analyses, respectively, performed on one of the samples from mine M. Each sample is associated with summary tables of this type. The throttle number designations 18, 17, 16, and 14 correspond to the BAHCO size cuts noted above, with throttle 18 containing the smallest particles and throttle 14 the largest. Table I indicates that the initial pre-BAHCO-separated sample was characterized for size by the BAHCO size distribution data (weights of four fractions) and by microscopy. Each of the four size fractions were sized by optical microscopy, and all but the fraction containing the smallest sizes were sized by sedimentation analysis. Results of particle size analyses are expressed in terms of the interquartile range because size distributions did not adhere to any theoretical distribution. As anticipated, there is little variation in density between particle size fractions, but large increases occurred in total surface area and projected area as particle size decreased.

Table II reveals increasing free silica content with decreasing particle size fraction within the respirable size range. There are large ash and carbon content variations for this sample in the respirable size range. In the trace metal category, aluminum content increases with decreasing particle size, as do zinc and iron, but calcium decreases.

Tables III and IV present similar results for one sample from mine R.

Table V is a summary of statistical testing performed to illustrate the procedure

TABLE III
Results of Physical Analysis of Four Fractions of Respirable Coal Mine Dust Collected by Horizontal Elutriator in Mine R and Separated by BAHCO (Type R-2 Coal)

| BAHCO Throttle No. | 18 | 17 | 16 | 14 | Total Dust |
|---|---|---|---|---|---|
| Geometric mean $d_s$ by BAHCO ($\mu$m) | | | | | 2.95 |
| Lower quartile $d^{25}{}_s$ by BAHCO ($\mu$m) | | | | | 1.99 |
| Upper quartile $d^{75}{}_s$ by BAHCO ($\mu$m) | | | | | 4.80 |
| Geometric mean $d_p$ by microscopy ($\mu$m) | 2.40 | 2.82 | 5.95 | 8.00 | 3.40 |
| Lower quartile $d^{25}{}_p$ by microscopy ($\mu$m) | 1.65 | 2.15 | 3.90 | 5.70 | 2.35 |
| Upper quartile $d^{75}{}_p$ by microscopy ($\mu$m) | 3.12 | 3.65 | 8.20 | 10.30 | 4.30 |
| Geometric mean $d_s$ by sedimentation balance ($\mu$m) | | | | | |
| Lower quartile $d^{25}{}_s$ by sedimentation balance ($\mu$m) | | | | | |
| Upper quartile $d^{75}{}_s$ by sedimentation balance ($\mu$m) | | | | | |
| Random projected area by light extinction (cm$^2$/gm) | $6.57 \times 10^3$ | $5.35 \times 10^3$ | $4.58 \times 10^3$ | $3.58 \times 10^3$ | |
| Total surface area by BET (cm$^2$/gm) | $7.93 \times 10^4$ | $6.17 \times 10^4$ | $4.76 \times 10^4$ | $4.31 \times 10^4$ | |
| Density by air pycnometry (gm/cm$^3$) | 2.33 | 2.25 | 2.21 | 2.31 | |

TABLE IV
Results of Chemical Analysis of Four Fractions of Respirable Coal Mine Dust Collected by Horizontal Elutriator in Mine R and Separated by BAHCO
(Type R-2 Coal)

| BAHCO Throttle No. | Substance | 18 | 17 | 16 | 14 | Total Dust |
|---|---|---|---|---|---|---|
| Free silica analysis (weight %) | $SiO_2$ | 2.6 | 2.7 | 2.7 | 2.2 | |
| | Ash | 43.8 | 39.5 | 38.1 | 41.3 | |
| Proximate analysis (weight %) | Moisture | | | | | |
| | Volatile matter | | | | | |
| | Fixed carbon | | | | | |
| | Ash | | | | | |
| Ultimate analysis (weight %) | Carbon | 39.9 | 46.3 | 46.4 | 45.1 | |
| | Hydrogen | — | — | — | — | |
| | Sulphur | — | 0.6 | 0.3 | — | |
| | Nitrogen | | | | | |
| | Oxygen | | | | | |
| | Ash | | | | | |
| Trace element analysis (weight %) | Aluminum | 2.6 | 4.0 | 2.8 | 2.8 | |
| | Barium | 0.07 | 0.05 | 0.07 | 0.07 | |
| | Beryllium | 0.003 | 0.003 | 0.003 | 0.001 | |
| | Bismuth | 0.04 | 0.02 | 0.03 | 0.27 | |
| | Cadmium | 0.007 | 0.005 | 0.006 | 0.007 | |
| | Calcium | — | — | — | — | |
| | Chromium | — | — | — | — | |
| | Cobalt | <0.01 | <0.02 | <0.01 | <0.01 | |
| | Copper | 0.45 | 0.28 | 0.03 | 0.10 | |
| | Iron | 0.80 | 0.83 | 1.30 | 0.85 | |
| | Lead | 0.08 | 0.06 | 0.06 | 0.03 | |
| | Lithium | 0.24 | 0.35 | 0.11 | 0.03 | |
| | Magnesium | 0.45 | 0.47 | 0.11 | 0.17 | |
| | Manganese | 0.01 | 0.01 | 0.02 | 0.015 | |
| | Nickel | 0.001 | 0.001 | 0.005 | 0.001 | |
| | Potassium | 0.7 | 0.4 | 1.3 | 0.2 | |
| | Silver | 0.04 | 0.02 | 0.03 | 0.04 | |
| | Sodium | 0.11 | 0.18 | 0.18 | 0.36 | |
| | Strontium | 0.09 | 0.03 | 0.09 | 1.5 | |
| | Vanadium | — | — | — | — | |
| | Zinc | 0.03 | 0.03 | 0.17 | 0.14 | |

TABLE V
Differences between Total Surface Area ($cm^2/gm$), of Particle Size Fractions for Three Samples of Respirable Coal Mine Dust from Coal Mine R

| BAHCO Throttle No. | Arithmetic Mean Total Surface Area ($cm^2/gm$) of Three Samples ($\bar{x}$) | Standard Deviation (S.D.) | Range of Total Surface of Three Samples | Percent Difference between Upper or Lower Limit of Range and Arithmetic Mean of Three Samples | Test of Significant Difference[a] |
|---|---|---|---|---|---|
| 18 | $8.61 \times 10^4$ | $8.35 \times 10^3$ | $7.93 \times 10^4$–$9.54 \times 10^4$ | 10.8 | N.S. |
| 17 | $6.93 \times 10^4$ | $6.73 \times 10^3$ | $6.17 \times 10^4$–$7.46 \times 10^4$ | 10.9 | N.S. |
| 16 | $5.28 \times 10^4$ | $5.30 \times 10^3$ | $4.76 \times 10^4$–$5.82 \times 10^4$ | 10.23 | N.S. |
| 14 | $4.51 \times 10^4$ | $2.23 \times 10^3$ | $4.31 \times 10^4$–$4.75 \times 10^4$ | 5.32 | N.S. |

[a]Test of significance indicates if $(\bar{x} - x)$ is $< \pm 2$ S.D.

used to determine if the properties summarized in Tables I through IV for two samples differ significantly between samples collected in each mine. Thus, for each physical property or result of chemical analysis for each size fraction, a mean value and standard deviation for three samples collected in a mine were obtained. Table V indicates that, at the 95% confidence level (±2 S.D.), the four particle size fractions of samples collected in mine R were associated with the same specific surface area. This finding was representative of results of this type of testing when it was applied to intersample variations in properties of samples collected in mines M and R. The only particle parameter showing significant variations between samples from the same mine was that of particle size, as measured by the mean size in each particle size fraction. Therefore, we concluded that at our sites of sampling, which were relatively close to the face, there are variations in size of the airborne dust, variations which may be damped further from the face.

### *Variations in Physical and Chemical Properties of Respirable Dust Samples Collected in Different Mines*

The procedures described above established average values and dispersion of these values for three respirable dust samples in

TABLE VI

Differences between Total Surface Areas ($cm^2/gm$) of Three Samples of Respirable Coal Mine Dust from Coal Mine M and Coal Mine R

| BAHCO Throttle Number | Arithmetic Mean Total Surface Areas of Three Coal Mine Samples from Coal Mine M ($\bar{x}_1$) | Arithmetic Standard Deviation of Three Coal Mine Samples from Coal Mine M ($s_1$) | Arithmetic Mean Total Surface Areas of Three Coal Mine Samples from Coal Mine R ($\bar{x}_2$) | Arithmetic Standard Deviation of Three Coal Mine Samples from Coal Mine R ($s_2$) | Test of Significance* |
|---|---|---|---|---|---|
| 18 | $2.03\times10^5$ | $4.38\times10^4$ | $8.61\times10^4$ | $8.35\times10^3$ | S. |
| 17 | $1.60\times10^5$ | $4.16\times10^4$ | $6.93\times10^4$ | $6.73\times10^3$ | S. |
| 16 | $1.06\times10^5$ | $8.70\times10^3$ | $5.28\times10^4$ | $5.30\times10^3$ | S. |
| 14 | $7.95\times10^4$ | $1.10\times10^4$ | $4.51\times10^4$ | $2.23\times10^3$ | N.S. |

$$*t = \frac{(\bar{x}_1 - \bar{x}_2) - (\mu_1 - \mu_2)}{\sqrt{(s_1^2/n_1) + (s_2^2/n_2)}} \text{ with } \nu \text{ degrees of freedom, where } \nu = \frac{\left(\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}\right)^2}{\frac{(s_1^2/n_1)^2}{n_1 - 1} + \frac{(s_2^2/n_2)^2}{n_2 - 1}}$$

TABLE VII

Differences between Free Silica Analysis (Weight %) of Three Samples of Respirable Coal Mine Dust From Coal Mine M and Coal Mine R

| BAHCO Throttle Number | Arithmetic Mean Free Silica (weight %) of Three Coal Mine Samples from Coal Mine M ($\bar{x}_1$) | Arithmetic Standard Deviation of Three Coal Mine Samples from Coal Mine M ($s_1$) | Arithmetic Mean Free Silica (weight %) of Three Coal Mine Samples from Coal Mine R ($\bar{x}_2$) | Arithmetic Standard Deviation of Three Coal Mine Samples from Coal Mine R ($s_2$) | Test of Significance* |
|---|---|---|---|---|---|
| 18 | 6.43 | 1.24 | 2.47 | 0.15 | S. |
| 17 | 6.68 | 1.58 | 2.87 | 0.38 | S. |
| 16 | 6.01 | 1.69 | 2.77 | 0.31 | N.S. |
| 14 | 5.24 | 0.37 | 2.77 | 0.74 | S. |

$$*t = \frac{(\bar{x}_1 - \bar{x}_2) - (\mu_1 - \mu_2)}{\sqrt{(s_1^2/n_1) + (s_2^2/n_2)}} \text{ with } \nu \text{ degrees of freedom, where } \nu = \frac{\left(\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}\right)^2}{\frac{(s_1^2/n_1)^2}{n_1 - 1} + \frac{(s_2^2/n_2)^2}{n_2 - 1}}$$

mine R and three samples in mine M. We then proceeded to test for whether or not significant differences between each of the particle parameters existed for samples collected in mines M and R. Table VI summarizes the statistical procedure used by offering as an example the test for whether or not the total surface areas of the respirable dust size fractions differ between mine M and mine R samples. It can be seen that, on the basis of the 95% confidence level selected to test the significance of the difference between the arithmetic mean values of this parameter for each size fraction, all but the largest particle size fraction differ significantly in samples colletced in the two mines. Table VII also illstrates the procedure and shows that free silica content of three of the four particle size fractions differed significantly between samples collected in mine M and mine R, respectively.

In general, respirable dust samples from mines M and R showed remarkably small differences between physical and chemical properties associated with the BAHCO-separated particle size fractions. However, the purpose of this paper was to illustrate the types of collection procedures and analytical methods believed to be necessary if valid epidemiological methods are to be used to determine which respirable dust properties are associated with high incidence of coal workers' pneumoconiosis in United States mines. In this regard, mines M and R are specimen mines employed to demonstrate the procedures to be utilized with respirable dust characterization in other United States mines. Initially, dust will be collected in a single mine in each seam. However, the methodology could be extended to include sample collection and analysis in each mine if financial considerations permit such extension.

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## American Industrial Hygiene Conference

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